Intercomparison of Permittivity Measurements Using the Transmission/Reflection Method in 7-mm Coaxial Transmission Lines

Eric J. Vanzura, James R. Baker-Jarvis, John H. Grosvenor, and Michael D. Janezic

Abstract— Broadband permittivity measurements made by eleven organizations using the transmission/reflection (T/R) method are compared to high-accuracy cavity resonator results. T/R accuracy is less than 10% for $\epsilon_r'<15$, and the smallest measureable loss factor is $\epsilon_r''\approx0.05$. Uncertainty caused by the air gaps between the specimen and the inner and outer conductors is the largest contributor to the overall uncertainty. Compared to other dimensional measurement methods, physical measurement of specimen bore and outer diameters yield the most accurate gap corrections.

I. INTRODUCTION

THE transmission/reflection (T/R) coaxial line method is frequently used by those interested in dielectric and magnetic properties measurements at radio and microwave frequencies, but currently suffers from a lack of standard procedures, algorithms, and materials. To aid in standardizing the T/R method, the National Institute of Standards and Technology (NIST) organized a nationwide intercomparison of permittivity measurements in the 50 MHz to 18 GHz frequency range. The T/R method involves calculation of complex relative permittivity ($\epsilon_r^* = \epsilon_r' - j \epsilon_r''$) and permeability (μ_r^*) from transmitted and reflected scattering parameters measured by a network analyzer. This intercomparison focuses on permittivity measurements only.

Three sample kits were circulated among eleven participating organizations. Each kit was circulated to at least three different participants. All three sample kits contain four materials with ϵ'_r ranging from approximately 6.8 to 17. Sample kit 3 contains a fifth, higher-permittivity specimen with $\epsilon_r' \approx 50$. The materials which make up the specimens were not disclosed to the intercomparison participants, and are summarized in γ Table I. To maintain the anonymity of participants' results, each organization has been assigned a letter code (A through J). A legend key is given in Table II to match symbol types, sample kit numbers, data-reduction algorithm types, and air-gap estimation methods. Along with a sample kit, each participant was sent a set of measurement guidelines and a measurement data sheet. Measurement guidelines included standard equipment specifications and a recommended procedure. The measurement data sheet was used to record relevant information including ambient conditions, network analyzer configuration, specimen holder dimensions, and test result file

Manuscript received October 5, 1993; revised December 20, 1993. The authors are with the Electromagnetic Fields Division, National Institute of Standards and Technology, Boulder, CO 80303-3328 USA. IEEE Log Number 9404631.

TABLE I
MATERIAL COMPOSITION AND PERMITTIVITIES OF INTERCOMPARISON
MATERIALS. MATERIAL 5 WAS CONTAINED ONLY IN KIT 3 AND NOT
IN KITS 1 AND 2. PERMITTIVITIES ARE FROM MEASUREMENTS MADE
USING THE NIST CYLINDRICAL CAVITY RESONATOR AT 10 GHz

Material	Composition	ϵ_r'	ϵ_r''
1	High-purity alumina	9.97	0.00039
2	Lead-oxide glass (NIST SRM 709)	11.78	0.126
3	Soda-lime glass (NIST SRM 710a)	6.82	0.049
4	Magnesium titanate ceramic	16.05	0.0014
5	Barium titanate ceramic	50.0	0.020

TABLE II
INTERCOMPARISON PARTICIPANTS LEGEND KEY GIVING SYMBOL
TYPES, SAMPLE KIT NUMBERS, DATA-REDUCTION ALGORITHMS,
AND AIR-GAP ESTIMATION METHODS. ALGORITHM AIR-GAP

Participant	Symbol	Kit	Algorithm	Air-Gap
Code	Type	Number	Туре	Estimation Method
A	-0-	1	Explicit	No gap correction
В		1	Explicit	No gap correction
C	-0-	1	EPSMU3	NIST provided
D		2	EPSMU3	Mechanically measured
E	+	2	Internal	Travelling microscope
F	-	2	Internal	Gage pins
G	-	3	Internal	NIST provided
Н	+	3	EPSMU3	Feel
I	-4-	3	EPSMU3	NIST provided
J	-4-	3	Internal	Stereo microscope
NIST T/R	-×-	3	EPSMU3	Mechanically measured
NIST	-	NA	NA	NA
Resonator	₹.			

names. Dielectric or conductive pastes or fillers to mitigate the effects of air gaps were forbidden. Participants were free to use any data-reduction algorithm and air-gap estimation method.

II. INTERCOMPARISON SPECIMENS

The five materials given in Table I are of interest to the NIST Electromagnetic Properties of Materials Program as possible dielectric reference materials. The inclusion of these materials in this intercomparison does not imply an endorsement by NIST. Reference materials to be distributed by NIST are first purchased from manufacturers in bulk quantities. The properties of these bulk materials are then thoroughly tested before NIST distribution to purchasers. This certification and distribution process helps to prevent uncharacterized batch-

Material	Specimen	Measured by	$_{ m Length}$	Inner	Outer
	Code	Organization	(mm)	Diameter (mm)	Diameter (mm)
1	1al	A, B, C	11.855	3.0633 ± 0.0134	6.9867 ± 0.0052
	2al	D, E, F	11.854	3.0593 ± 0.0106	6.9901 ± 0.0079
	3al	G, H, I, J	11.844	3.0624 ± 0.0068	6.9851 ± 0.0067
2	1lg	A, B, C	11.856	3.0727 ± 0.0079	6.9892 ± 0.0057
	2lg	D, F	11.562	3.0515 ± 0.0040	6.9922 ± 0.0061
	3lg	Broken	11.583	3.050 ± 0.012	6.975 ± 0.012
	4lg	E, G, J	11.722	3.0539 ± 0.0086	6.9732 ± 0.0069
	5lg	H	11.374	3.0547 ± 0.0023	6.9772 ± 0.0076
	6lg	I	9.738	3.0569 ± 0.0054	6.9791 ± 0.0083
3	1sg	A, B, C	11.833	3.0609 ± 0.0079	6.9794±0.0079
	2sg	D, E, F	11.847	3.0605 ± 0.0104	6.9936 ± 0.0050
	3sg	G, H, I, J	11.838	3.0575 ± 0.0067	6.9925 ± 0.0056
4	1ds	A, B, C	19.720	3.0491 ± 0.0018	6.9886 ± 0.0018
	2ds	D, E, F	19.720	3.0491 ± 0.0026	6.9921 ± 0.0034
	3ds	G, H, I, J	19.728	3.0490 ± 0.0020	$6.9888 \!\pm\! 0.0025$
5	3df	GHIJ	25 532	3 0548+0 0022	6 9875+0 0021

TABLE III
INTERCOMPARISON SPECIMEN DIMENSIONS MEASURED AT NIST USING A COORDINATE MEASURING MACHINE (CMM)

to-batch variations. NIST also has bulk quantities of other dielectric and magnetic materials which are not included in this intercomparison.

The coaxial alumina specimens were machined by NIST from a previously tested 60-mm diameter cavity resonator specimen. NIST also machined coaxial specimens from bulk quantities of the lead-oxide and soda-lime glasses. These glasses are standard reference materials (SRM) used for high-temperature viscosity calibrations and are not considered dielectric reference materials. All coaxial lead-oxide glass specimens were machined from the same block. These SRM 709 lead-oxide glass coaxial specimens were prepared from a different block than the 60-mm diameter cavity resonator specimen. Coaxial specimens machined from the soda-lime glass SRM 710 a were prepared from the same block as the cavity resonator specimen. Separate cavity and coaxial specimens of materials 4 and 5 were pressed, fired, and machined by the manufacturer from the same batches. As we will see, differences in specimen preparation may cause variations in permittivities of specimens with identical composition but different geometries.

NIST provided estimated specimen length, and inside and outside diameters to all participants. A coordinate measuring machine (CMM) with $\pm~0.0015$ mm measurement uncertainty was used to measure specimen diameters. These NIST-measured specimen dimensions are given in Table III. The specimen inside and outside diameter estimates supplied to participants were calculated as the average of diameter measurements taken every millimeter along each specimen's length. The diameter uncertainties stated in Table III are the standard deviations of diameter measurements along the specimen length. As will be discussed later, many participants used their own dimensional estimation methods.

This intercomparison was completed with all alumina, sodalime glass, magnesium titanate, and barium titanate specimens intact. As Table III shows, several lead-oxide glass specimens, being brittle, were broken during the course of the intercomparison.

III. MEASUREMENT CONSIDERATIONS

A. Data-Reduction Algorithms

A commonly implemented algorithm [1]-[3] finds an explicit solution for permittivity (and permeability) from measured scattering parameters. This method results in an ill-conditioned set of equations when the specimen length is equal to an integral number of half-wavelengths inside a low loss specimen. Participants A and B used this explicit algorithm to calculate permittivity, and their results are typical. The explicit algorithm yields very poor precision, especially at the higher frequencies, and the effects of the half-wavelength instability of the explicit solution is obvious. This half-wavelength problem has been customarily avoided through the use of short specimens. However, short specimens often lead to additional uncertainties in specimen length, alignment, location, and geometry that are difficult to quantify or correct.

An iterative algorithm developed by Baker-Jarvis at NIST [4]-[6] and other algorithms developed internally by others [7] companies yield much more precise permittivity estimates compared to the explicit algorithm. The NIST algorithm employs a Newton-Raphson iterative method to solve for permittivity (and permeability, if desired). An initial estimate of permittivity (and permeability) is required to find the correct solution. Low-frequency extrapolation, group delay, and other initial-estimate root-finding techniques can be applied.

B. Air-Gap Corrections

Accurate knowledge of air-gap dimensions is fundamental to the proper determination of material characteristics using the T/R method. Dimensional measurements of both the specimen and the specimen holder's inner and outer diameters must be made in order to estimate air gap sizes. Table II lists the specimen-dimension estimation methods used by participants. Uncertainty in the air gap dimensions is usually the primary contributor to overall measurement uncertainty. Baker-Jarvis

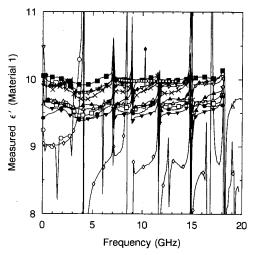


Fig. 1. Measured relative permittivity (ϵ'_r) of material 1.

[4] discusses measurement uncertainty in detail and it will not be repeated here. Participants were not asked to provide details regarding their air-gap correction methods. However, many participants, including NIST, use a coaxial capacitor model. The coaxial capacitor consists of two air layers and one specimen dielectric layer. We assume the air gaps to be uniform between the specimen and the inner and outer conductors, and treat the system as three capacitors in series. Since the electric field is greatest near the center conductor, the relative correction for the inner-conductor air gap is greater than that of the outer-conductor gap. For a given air-gap, the total correction significantly increases as specimen permittivity increases [4, p. 113]. The results of this intercomparison demonstrate that, as long as the dimensions of the specimen and specimen holder are accurately known, this uniform air gap model yields good results for low to moderate permittivity values and small gaps.

C. Reducing the Need for Air Gap Corrections

The coaxial specimens circulated in this intercomparison were very accurately machined so as to minimize air gaps. As the results show, even small air gaps lead to large corrections, especially for high permittivity materials. Some of the participants routinely use some means to reduce the air gap correction. For example, one participant uses a specially designed coaxial holder and custom machined specimens and does not usually apply an air gap correction. The details of this specimen holder are not known. This participant submitted gap-corrected results from measurements using a precision 7mm diameter beadless air line. Many participants, including NIST, routinely make measurements in larger diameter coaxial specimen holders. In this situation, air gaps can be held small relative to coaxial line diameters. This reduces the relative sizes of the air gaps, and therefore reduces the magnitude of the air gap correction. One disadvantage to these large diameter coaxial systems is their limitation to lower frequencies.

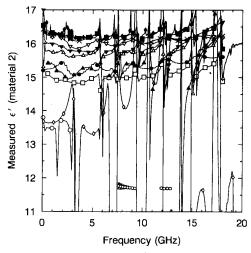


Fig. 2. Measured relative permittivity (ϵ'_r) of material 2.

Rectangular waveguides can also be used for materials measurements in a given frequency band. Rectangular waveguides require much smaller gap corrections than coaxial systems that operate at similar frequencies because of differences in electric field distributions. Additionally, rectangular specimens can be machined with smaller tolerances than coaxial specimens. This reduces the size of the air gaps and thus reduces the magnitude of the correction. The need for air gap corrections can be eliminated by using conductive pastes or high dielectric constant fillers. This approach can be especially useful for measurements of very high permittivity materials. Although the use of fillers was forbidden in this intercomparison, most participants occasionally use such means to fill air gaps. Special care must be taken that the filler does not contaminate the specimen ends. A positive bias in measured dielectric loss (ϵ_r'') always arises from this approach either because the paste or filler is a lossy material or because of specimen contamination. Additionally, the emulsifier can migrate into porous specimens and affect both ϵ'_r and ϵ''_r results by modifying line impedance.

IV. INTERCOMPARISON MEASUREMENT RESULTS

Measurement results from the participating organizations are shown in Figs. 1–10. An iterative method is used by the EPSMU3 software supplied by NIST. Most participants who used their own internally developed algorithms also applied iterative techniques. Some participants did not provide any details regarding their data-reduction algorithms. To provide a measurement reference, the figures also include results and uncertainties from 8 to 12 GHz cavity resonator measurements made by NIST on 60-mm diameter disk-shaped specimens. A description of this cylindrical cavity resonator may be found in [8].

A. Dielectric Constant (ϵ'_r) Measurements

With gap corrections, most participants obtained agreement with cylindrical cavity resonator measurements of ϵ'_r within

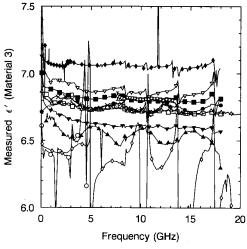


Fig. 3. Measured relative permittivity (ϵ'_r) of material 3.

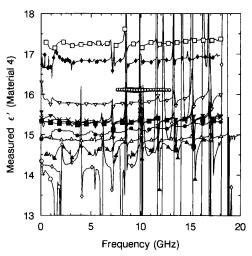


Fig. 4. Measured relative permittivity (ϵ'_r) of material 4.

10%. Participant D made their own dimensional measurements using a CMM. The results of Participant D closely agree with NIST coaxial measurements. With the exception of the lead-oxide glass, NIST and Participant D's results are consistently within 5% of the values measured using the cavity resonator. As will be discussed later, the good results of participant D can be attributed to having made physical measurements of specimen and air line dimensions. The results of those participants that used NIST-provided specimen dimensions also agree closely with NIST measurements. The observed differences in these participants' measurements is probably attributable to unknown variations in specimen holder diameters.

Participants A and B used an explicit algorithm to calculate permittivity. Because Participant A typically measures short specimens, their algorithm did not include a root-selection routine. For higher frequencies at which the specimen is longer than a half wavelength, Participant A calculated permittivities

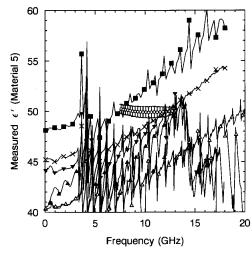


Fig. 5. Measured relative permittivity (ϵ'_r) of material 5.

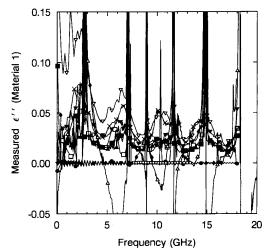


Fig. 6. Measured loss factor (ϵ'_r) of material 1.

TABLE IV

CENTER CONDUCTOR AND OUTER CONDUCTOR DIAMETERS
OF NIST AND PARTICIPANT D SPECIMEN HOLDERS,

	Center Conductor Diameter (mm)	Outer Conductor Diameter (mm)
NIST	3.041 60	6.9968
Participant D	3.038 88	7.0014

that are off the scale of the given figures. Participants A and B also did not apply gap corrections. This results in a negative bias that is easiest to discern at the lower frequencies, where their permittivity results are most stable. To apply a gap correction, we must know the diameters of both the specimen and the specimen holder. The diameters of the 7-mm coaxial specimen holders used by NIST and by participant D are given in Table IV, so the magnitude of gap corrections for a given gap and permittivity can be gauged.

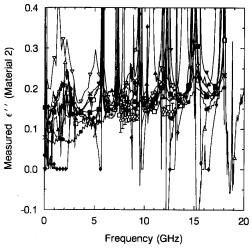


Fig. 7. Measured loss factor (ϵ'_r) of material 2.

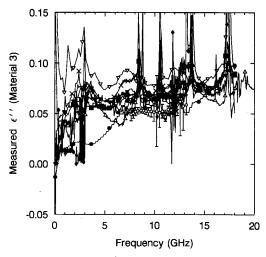
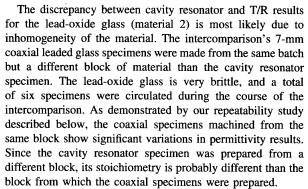


Fig. 8. Measured loss factor (ϵ'_r) of material 3.



The results of Participant I vary with frequency. This behavior can usually be attributed to an error in which either or both the specimen or specimen holder lengths were entered

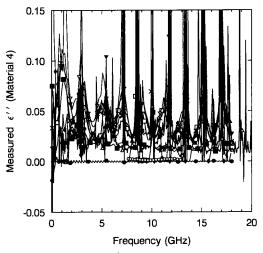


Fig. 9. Measured loss factor (ϵ'_r) of material 4.

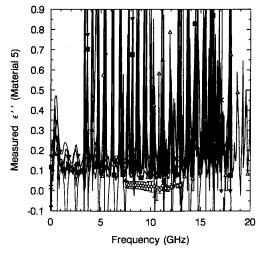


Fig. 10. Measured loss factor (ϵ'_r) of material 5.

incorrectly into the data reduction routine. Also, a data entry error might be attributed to Participants C and E's results for material 4 in which the estimated air gaps were too large. We can see that Participant E's ϵ'_r results are on the high side of the range of results, while Participant I is consistently on the low side.

The permittivity of material 5 was particularly difficult to measure because it has a very high permittivity and because it is nearly twice as long as the other specimens. High permittivity specimens are very sensitive to air gap corrections and excite higher-order modes. All five organizations that submitted permittivity data for material 5 show increasing ϵ'_r with increasing frequency. Propagation of higher order modes inside the specimen might explain this increasing measured permittivity and is presently a subject of research at NIST. The phase velocities of higher-order TE and TM modes

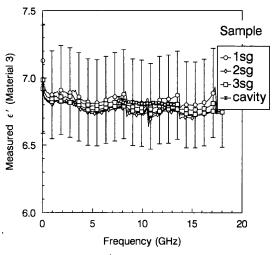


Fig. 11. Permittivity results (ϵ'_r) from repeated measurements of NIST SRM 710a soda-lime glass (material 3). Results are shown for all three soda-lime glass specimens. Large error bars correspond to estimated T/R-method uncertainties for specimen 1 sg. Small error bars are estimated uncertainties for cavity resonator measurements of a disk-shaped specimen.

propagating inside the specimen are slower than the phase velocity of the assumed dominant TEM mode. These lower phase velocities can make the specimen appear to have a higher permittivity as more non-TEM modes are excited in the specimen region. Another explanation under investigation at NIST is that the commonly assumed three-capacitor model used in the air-gap correction formulation breaks down for high-permittivity materials.

B. Loss Factor (ϵ_r'') Measurements

All of the dielectric specimens measured in this intercomparison are low- to medium-loss materials. Low-loss materials challenge the capability of the T/R technique in that sources of systematic error become readily apparent. The explicit algorithms used by Participants A and B yield unstable $\epsilon_n^{"}$ results, so their data have not been included in the figures to improve intelligibility. As shown in Figs. 6-10, most participants returned measurements that have large ϵ_r'' variations. The frequencies at which the most severe deviations occur correspond to resonances in which the specimen length is an integral multiple of one half-wavelength of the propagating mode. Very slight imperfections in specimen geometry will cause significant changes in the quality factor of these resonances and therefore effect significant deviations in the calculated loss factor at these frequencies. Participant F provides a notable exception. Their measurement results were calculated using a proprietary algorithm that appears to be stable throughout the measured frequency range. No hypothesis is offered for the well-behaved nature of Participant F's loss factor results because we have no additional information concerning their algorithm.

V. REPEATABILITY MEASUREMENTS

Several measurements to demonstrate repeatability and specimen-to-specimen variations were made by NIST during

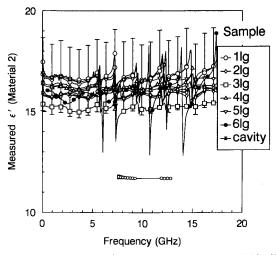


Fig. 12. Permittivity results (ϵ_r') from repeated measurements of NIST SRM 709 lead-oxide glass (material 2). Results are shown for all six lead-oxide glass specimens circulated during the intercomparison. Large error bars correspond to estimated T/R-method uncertainties for specimen 1 lg. Small error bars are estimated uncertainties for cavity resonator measurements of a disk-shaped specimen.

the intercomparison on the all 7-mm specimens. For materials other than the lead-oxide glass, permittivity results showed insignificant differences between specimens relative to the estimated uncertainty of each specimen. Permittivity measurement results shown in Fig. 11 for the soda-lime glass are typical. Fig. 12 shows the results of repeated measurements made by NIST on the lead-oxide glass specimens. Measurements of the same specimen yield repeatable results, but specimen-to-specimen differences in permittivity can be discerned from the fact that permittivity of one specimen is often outside the estimated uncertainty calculated for other lead-oxide glass specimens. As such, the brittleness and inhomogeneity of the lead-oxide glass material makes it undesirable as a reference dielectric material.

VI. CONCLUSION

This intercomparison of measurements of the dielectric constants of 7-mm diameter coaxial specimens demonstrates that iterative algorithms to compute permittivity from measured scattering parameters are superior to the explicit algorithms that were commonly used before the mid-1980's. Results calculated using explicit algorithms that simultaneously calculate permittivity and permeability are unstable, especially at frequencies where wavelength of the TEM mode inside the specimen is an integral multiple of one half-wavelength. In comparison, ϵ'_r results from iterative algorithm calculations yield good stability, with some frequency-dependent fluctuations perhaps due to specimen imperfections or errors in data entry. Dielectric loss ϵ_r'' measurement results are much less precise. Half-wavelength resonances, specimen imperfections, and lack of scattering parameter precision combine to make dielectric loss measurements untenable for $\epsilon_r''/\epsilon_r' \lesssim 0.05$ for low to medium dielectric constants.

Those participants who used the iterative algorithm for the first four materials generally agree within 10% of each other. Cavity resonator and coaxial specimens made from materials 1 and 3 were prepared from the same block, and cavity resonator and T/R measurements of these three materials agree within 10%. Coaxial measurement of material 2 also fall within 10% of each other, but significant variations in measured permittivity have been observed between coaxial specimens. In addition coaxial measurements of material 2 are significantly higher than cavity resonator results. The cavity specimen was prepared from a different block. This material is inhomogeneous and should be considered undesirable as a reference dielectric material. Coaxial and cavity specimens of material 4 were prepared individually. All three coaxial specimens appear to have substantially equivalent permittivities, but there appears to be a measureable difference between the cavity specimens and the coaxial specimens in which the cavity specimens have higher permittivity. A similar result occurs for material 5; all participants' ϵ'_r results are lower than the cavity resonator results from two different cavity specimens. In addition, material 5 shows a trend in which measured ϵ'_r increases as frequency increases. Similar results have been observed for other high dielectric constant materials, and the cause is most likely due to increased propagation of non-TEM modes inside the specimen as frequency is increased.

A correction to permittivity results is necessary due to air gaps between the specimen and the specimen holder. The presence of air gaps biases permittivity results low. This correction becomes more significant for higher permittivities to the point of limiting the usefulness of this technique. The uncertainty in the air-gap correction is the primary error source of the T/R method. Participants used coordinate measuring machines, microscopes, gage pins and subjective "feel" methods to estimate air gaps. Results from participants who used a coordinate measuring machine (NIST and Participant D) to estimate air gaps are somewhat better than the results from participants that used other methods.

ACKNOWLEDGMENT

The authors thank the following organizations for donating their time and efforts in participating in this measurement intercomparison: 3M Company, St. Paul, MN; Boeing Defense and Space Group, Seattle, WA; Dow-Corning Corp., Midland, MI; Georgia Tech. Research Institute, Atlanta, GA; Grumman Aircraft Corp., Bethpage, NY; Hewlett-Packard Company, Santa Rosa, CA; Lockheed Research Laboratories, Palo Alto, CA; McDonnell Aircraft Co., St. Louis, MO; Naval Research Laboratory, Washington, DC; and SRI International, Palo Alto, CA. They would like to thank G. Sherwood for dimensional measurement work.

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